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Catalytic pyrolysis of sugarcane bagasse by using microwave heating

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HIGHLIGHTS

- Mass reduction ratio and reaction rate can be increased by catalyst addition.
- Different metal oxides can provide different product distributions.
- There could be several secondary reactions to alter the product distribution.
- Catalyst addition can increase pre-exponential factor greatly.

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ABSTRACT

The aim of this study was to research the catalytic effects on the microwave pyrolysis of sugarcane bagasse and thus to discuss the reaction performance, product distribution, and kinetic analysis. With the addition of metal-oxides served as catalysts, reaction results such as mass reduction ratio and reaction rate increased, even the maximum temperature decreased. Adding either NiO or CaO slightly increased the production of H_2, while adding either CuO or MgO slightly decreased it. The addition of either CaO or MgO enhanced the gaseous production, and either NiO or CuO addition enhanced the liquid production. There could be several secondary reactions such as self-gasification and interactions among the gases originally produced during the pyrolysis stage to alter the composition of gaseous product and the final three-phase product distribution. The catalyst addition slightly increased the activation energy but greatly increased the pre-exponential factor.

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1. Introduction

In recent years, many methods have been researched and developed for the conversion of biomass into valuable biofuels and bio-based products. These methods can be generally divided into two categories: thermo-chemical and bio-chemical. Thermo-chemical conversion technologies primarily include combustion, gasification, and pyrolysis. Depending on the operating conditions, the pyrolysis processes can be divided into three subclasses: conventional pyrolysis, fast pyrolysis, and flash pyrolysis (Maschio et al., 1992). Compared with conventional heating, microwave (dielectric) heating can provide several benefits. In conventional heating methods, heat is transferred into materials by convection, conduction, and radiation to material surfaces. On the contrary, microwave energy is delivered directly into materials within an electromagnetic field. Therefore, energy is transferred by thermal gradients in the conventional heating, but microwave heating is the conversion of electromagnetic energy into molecular kinetic energy rather than heat transfer. The phenomenon of energy conversion can make the microwave heating more efficient. The difference between microwave and conventional heating can result in many potential advantages of using microwaves for the processing of materials such as that heat can be generated throughout the volume of the material, the energy transfer does not rely on diffusion of heat from the surfaces, and it is possible to achieve rapid and uniform heating of thick materials (Thostenson and Chou, 1999).

There have been several researches about the microwave pyrolysis of biomass feedstocks such as oil shale (El harfi et al., 2000), oil-palm stone (Guo and Lua, 2000), paper (Miura et al., 2001), plastic waste (Ludlow-Palafox and Chase, 2001), sewage sludge (Menendez et al., 2002), rock phosphate (Bilali et al., 2005), coffee hulls (Dominguez et al., 2007), wood (Chen et al., 2008), rice straw (Huang et al., 2010), corn stalk bale (Zhao et al., 2010), oil palm biomass (Salema and Ani, 2011), and automotive engine oil (Lam et al., 2010). According to these researches, the microwave pyrolysis of biomass-related feedstocks can produce various biofuels and bio-based products such as bio-oil, biochar, syngas, and hydrogen depending on factors including operating parameters, reaction atmosphere, and biomass characteristics (Huang et al., 2012). However, the reaction performance, productivity, and pilot- and

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full-scale data of the technology are still necessary to be further researched to promote its commercial applicability (Lam and Chase, 2012). Catalyst addition could be a practical way to promote the reaction performance, productivity, and so forth. Catalytic biomass pyrolysis is gaining increasing interest, and the use of catalysts or additives to improve the yield or quality of gaseous or liquid fuel from biomass is still in its infancy (Chen et al., 2008). Catalytic pyrolysis usually produces additional water and coke-solid residues and thus reduces the yield of organic phase in bio-oils. Some catalysts can improve the bio-oil yield, and chloride salts in particular simplify the chemical compositions of the resultant bio-oils and therefore improve the product selectivity of the pyrolysis process (Wan et al., 2009).

The goal of this study is to research catalytic effects on the microwave pyrolysis of sugarcane bagasse. Reaction performance and the characteristics and distribution of products are discussed.

2. Methods

2.1. Sugarcane bagasse

The sugarcane bagasse was obtained by the squeeze of red sugarcane which was cultivated in Nantou County, Taiwan. Before relevant tests, the sugarcane bagasse was shredded and sieved by a 60-mesh screen.

2.2. Metal-oxide catalysts

This study chose four metal oxides, NiO, CuO, CaO, and MgO, as catalysts for the microwave pyrolysis of sugarcane bagasse. The four metal oxides were chosen because they are cheap, easily obtained, and commonly used in petrochemical refinery and chemical synthesis processes. Compared with the X-ray diffraction (XRD) peaks of original metal oxides, the peaks of metal oxides after microwave heating did not shift. Therefore, it can be concluded that the microwave irradiation would not affect the properties of these metal oxides. The particle sizes of NiO, CuO, MgO, and CaO were 0.5–6, 1–4, 3–6 mm, and <20 mesh, respectively. Because the maximum adding quantity of the catalysts was only 10% of sugarcane bagasse sample weight, the mixing uniformity caused by the particle size could be negligible.

2.3. Experimental procedure

This study used a microwave processing device that can be seen elsewhere (Huang et al., 2010). Its irradiation manner was single mode (focused). The amount of sugarcane bagasse was 3–5 g in each experimental batch. The catalyst was added into the shredded and sieved sugarcane bagasse for the additive ratios of 3%, 5%, and 10% by weight. The mixed sample was filled in a quartz crucible, and then it was placed inside a quartz tube and precisely in the pathway of microwaves. In order to maintain anoxic circumstances and to purge out the vapor generated during microwave pyrolysis, inert gas (N_2) was purged into the system with a flow rate of 50 mL/min. After enough purging, the power supply of microwave device was turned on and switched to the designated microwave power level (500 W) for the designated processing time (30 min). Reflection microwave power levels were controlled to be less than 10% of incident ones during the whole experiment period. After passing through a condenser, the vapor generated by microwave pyrolysis was divided into two parts: liquid (condensable) and gas (incondensable). When the designate processing time was reached, the power supply was turned off and the carrier gas was shut down, and then the liquid and gas collectors were removed and sealed. After self-cooling down to room temperature, the solid residues remained in the crucible were removed and placed in a desiccator.

2.4. Product analysis

The analysis of gaseous product was carried out by a PerkinElmer AutoSystem XL gas chromatography-thermal conductivity detector (GC-TCD) with a column of Supelco Carboxen 1010 PLOT. The temperatures of injector, oven, and detector were 120, 100, and 150 °C, respectively. The flow rate of carrier gas (He/N_2) was 10 mL/min with a split of 25:1. The liquid product was analyzed by PerkinElmer TurboMass Gold gas chromatography/mass spectrometry (GC/MS) with a Supelco Equity-5 capillary column. The temperatures of injector and oven were 180 and 240 °C, respectively. The flow rate of carrier gas (He) was 10 mL/min with a split of 20:1. The calorific values of solid residues were determined by a CAL2K ECO calorimeter.

3. Results and discussion

3.1. Composition of sugarcane bagasse

The moisture fraction of the as-received sugarcane bagasse was about 2 wt%. The average calorific value of sugarcane bagasse was 17.98 MJ/kg. Most of the sugarcane bagasse was volatile matter and the fraction was about 97 wt%. The fractions of fixed carbon and ash content were only 0.12 and 2.85 wt%, respectively. The volatile matter refers to the components of sugarcane bagasse (excluding the moisture content) which are thermally liberated or decomposed in the absence of air or oxygen. The sugarcane bagasse was primarily composed of three elements, C, H, and O, whose fractions were about 45, 7, and 44 wt%, respectively. The extractives, hemicellulose, cellulose, and lignin fractions of sugarcane bagasse were about 7, 29, 50, 13 wt%, respectively. Thus, about 79 wt% of the sugarcane bagasse was composed of polysaccharides. Minerals such as SiO_2, Na_2O, Al_2O_3, and K_2O were the primary components in the ash content of sugarcane bagasse, and their fractions were about 88, 5, 3, and 2 wt%, respectively.

3.2. Temperature profile

The temperature profiles of sugarcane bagasse pyrolysis with and without catalyst addition are shown in Fig. 1. In the first minute, the reaction temperature slowly rose possibly due to the lack of enough electromagnetic energy to activate the bagasse. The temperature sensor delay could be neglected, because the sample temperature measured by a thermocouple was double checked by an infrared thermometer. Besides, it was observed that there was no water evaporation during the first minute of the experiments. After about 1 min, the bagasse was quickly heated at the rate of 62.9 °C/min (the average value from the start to the 8th min) or 128 °C/min (maximum). The maximum temperature was 532 °C at about 8th min. After the processing time of around 8 min, the reaction temperature remained approximately constant to the end of experiment (30 min processing). The addition of metal oxides would not affect the reaction temperature much and only lowered both the maximum temperature and heating rate slightly, as listed in Table 1. With catalyst addition, the average heating rate and the maximum temperature were 57.4–60.8 °C/min and 496–516 °C, respectively. Among these catalysts, the addition of CaO generally lowered the reaction temperature most, which may imply that CaO would influence the dielectric heating of reaction system most. The correlation coefficient of catalyst quantity and temperature was calculated to determine the correlation between the two factors. The quantity of either CuO or CaO

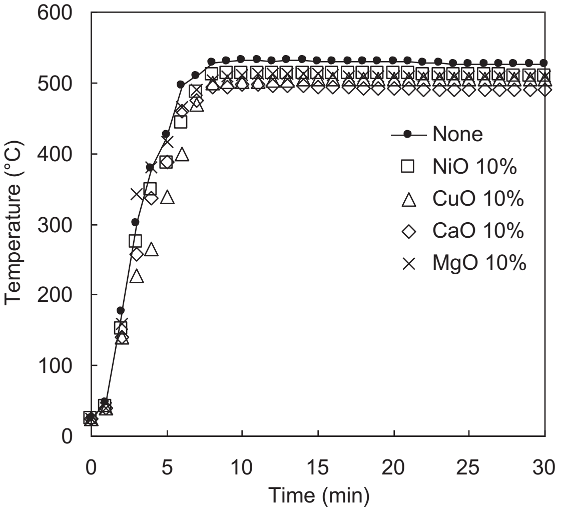

Fig. 1. The temperature profiles of microwave pyrolysis of sugarcane bagasse with and without catalyst addition.

Table 1
The maximum temperatures and heating rates of microwave pyrolysis of sugarcane bagasse.

Catalyst	Maximum temperature (°C)	Heating rate (°C/min)	
		Average[a]	Maximum
None	532	62.9	128.0
NiO 3%	505	59.7	100.9
NiO 5%	498	58.9	101.3
NiO 10%	514	60.8	122.7
CuO 3%	515	60.3	91.9
CuO 5%	496	57.9	97.1
CuO 10%	506	59.4	100.2
CaO 3%	496	58.4	116.7
CaO 5%	496	57.8	120.4
CaO 10%	497	58.6	116.4
MgO 3%	493	57.4	161.0
MgO 5%	516	60.1	193.3
MgO 10%	513	59.8	184.0

[a] The average value from the start to the 8th min.

had a moderate negative correlation while either NiO or MgO had a weak negative correlation with the reaction temperature.

3.3. Mass reduction ratio

Catalyst addition increased the mass reduction ratio of microwave pyrolysis of sugarcane bagasse, as listed in Table 2. Without any catalyst, the mass reduction ratio was 81.3%. After the catalyst was added, the mass reduction ratio became 82.7–86.9%. It is surprising that the catalyst addition lowered the reaction temperature but increased the mass reduction ratio. This may imply two possible advantages provided by catalyst addition: (1) the lower reaction temperature could decrease the possibility of system damage; (2) the higher mass reduction ratio could mean that there are more liquid and gas produced. Generally, compared with NiO and MgO, the addition of CuO and CaO could provide higher mass reduction ratios. This could be owing to the catalytic effect to convert more sugarcane bagasse into liquid and gaseous products. The quantity of either NiO or CaO had a strong positive correlation while CuO had a moderate positive correlation with the mass reduction ratio. The correlation between MgO quantity and the mass reduction ratio was negligible.

Table 2
The mass reduction ratios and calorific values of sugarcane bagasse after microwave pyrolysis.

Catalyst	Mass reduction ratio (%)	Calorific value (MJ/kg)
None	81.3	24.38
NiO 3%	82.7	23.71
NiO 5%	84.4	23.52
NiO 10%	84.6	22.84
CuO 3%	86.9	23.94
CuO 5%	85.1	23.61
CuO 10%	85.4	23.63
CaO 3%	85.9	23.55
CaO 5%	85.4	23.31
CaO 10%	86.9	22.94
MgO 3%	84.9	23.86
MgO 5%	83.5	23.81
MgO 10%	82.4	23.29

3.4. Calorific value of solid residues

Table 2 also lists the calorific values of solid residues. The calorific value was 24.38 MJ/kg without any catalyst, which was much higher than that of the original sugarcane bagasse (17.98 MJ/kg). The high calorific value shows the potential of solid residues to be regarded as a biofuel. The catalyst addition slightly lowered the calorific values, which were 22.84–23.94 MJ/kg. As above mentioned, the mass reduction ratios were increased when using catalysts. This may imply the existence of fixed-carbon content decomposition or volatilization due to severer pyrolysis, and thus the calorific values were decreased (Huang et al., 2008). Nevertheless, no matter with or without the catalysts, all the calorific values should be capable of replacing coal from the viewpoint of calorific value (Ullmann, 2003). Each of the quantity of NiO, CaO, and MgO had a very strong negative correlation ($p < 0.05$) with the calorific value of solid residues, while the correlation for CuO was strong negative.

3.5. Quantity of gaseous product

The gaseous product means the incondensable part of vapor produced by sugarcane bagasse pyrolysis via microwave heating. During the experiment, the flow rate of gaseous product was recorded every second by a digital-type flow meter. Fig. 2 shows the accumulated volume of gaseous product after 10, 20, and 30 min processing time when the quantities of the four catalysts were all 10 wt% of sugarcane bagasse sample. Without catalyst addition, about 78% of the gaseous product was collected within the first 10 min, and the total volume was about 2.4 L after

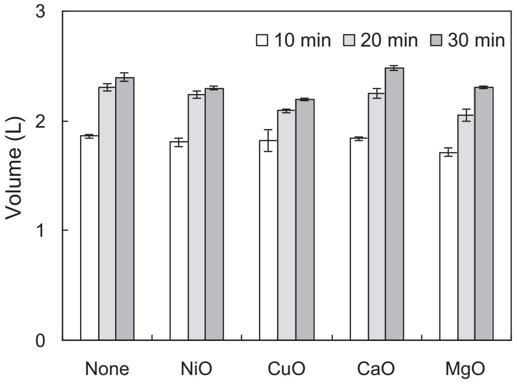

Fig. 2. The accumulated volume of gaseous product after 10, 20, and 30 min processing time (catalyst quantity: 10 wt%).

30 min processing time. Most of the gaseous product could be collected after 20 min, so only about 4% of the gas was produced during 20–30 min processing time. The addition of CaO increased the gas production (the total volume was about 2.5 L), while the other catalysts decreased the production. The total volumes of gaseous products under the addition of NiO, CuO, and MgO were about 2.3, 2.2, and 2.3 L, respectively.

3.6. Analysis of gaseous product

The gaseous products were analyzed to determine the volumetric concentrations of H_2, CH_4, CO, and CO_2, as shown in Fig. 3. Without catalyst addition, the average concentration of H_2, CH_4, CO, and CO_2 were 22.09, 2.69, 47.51, 15.05 vol%, respectively. This means that the syngas (H_2 + CO) content was about 70 vol%, and only about 13 vol% of the gaseous product was not determined. The undetermined part could be some light hydrocarbons such as C_2H_4 and C_2H_6 (Menendez et al., 2007) Adding either NiO or CaO increased the production of H_2 by about 1–3 vol%, while adding either CuO or MgO decreased it by about 3–6 vol%. The addition of the four catalysts all decreased the production of CH_4; and the more catalysts were used, the less CH_4 was produced. Only adding CuO increased the production of CO by about 1–3 vol% while the other catalysts decreased it. Similarly, only adding MgO increased the production of CO_2 by about 0.1–0.6 vol%, while the other catalysts decreased it.

By applying the ideal gas law, the molecules of H_2, CH_4, CO, and CO_2 produced with and without catalyst addition can be calculated, as listed in Table 3. The higher productions of H_2 and CO may imply that the addition of either NiO or CaO let the equilibrium of methane steam reforming (Xu and Froment, 1989) move toward the right-hand side of the reaction. In other words, adding either NiO or CaO can be helpful for the formation of syngas. As for CuO and MgO, adding either of them decreased the production of syngas. In fact, there was also less CH_4 and CO_2 generated when using the catalysts. This may imply that the addition of either CuO or MgO could inhibit the formation of the four gases. Otherwise, under the circumstance along with either catalyst, there would be a reaction like the Fischer–Tropsch synthesis that converted H_2 and CO into hydrocarbons such as alkanes (Schulz, 1999). Furthermore, the reduction of H_2 and CO could alter the equilibria of methane steam reforming, Boudouard reaction, and water–gas shift reaction. The Boudouard reaction is the gasification of carbon with CO_2 to produce CO (Jaber and Probert, 1999). The water–gas shift reaction is the interaction between the gases (Newsome, 1980):

$$CO + H_2O \leftrightarrow CO_2 + H_2 \quad (1)$$

Consequently, all the four gases were less produced to recover the equilibria of these gaseous reactions. This phenomenon should be induced by the existence of Fischer–Tropsch synthesis. Besides, according to Boudouard reaction, the consumption of CO would promote the decomposition of solid residues (char), which can be corresponding to the higher mass reduction ratios provided by the catalyst addition.

In addition to the microwave pyrolysis of sugarcane bagasse, there should be several secondary reactions including self-gasification and interactions among the gases originally produced during the pyrolysis stage. These reactions, which could be affected by catalyst addition, could change the composition of gaseous product and reduce the amount of solid residues. The decrease of solid residues means the increase of mass reduction. Therefore, the addition of catalyst could promote the reactivity of microwave pyrolysis.

4. Product distribution

The three-phase product distributions with and without catalyst addition are shown in Fig. 4. It can be seen that different catalysts led to different product distributions. Without catalyst

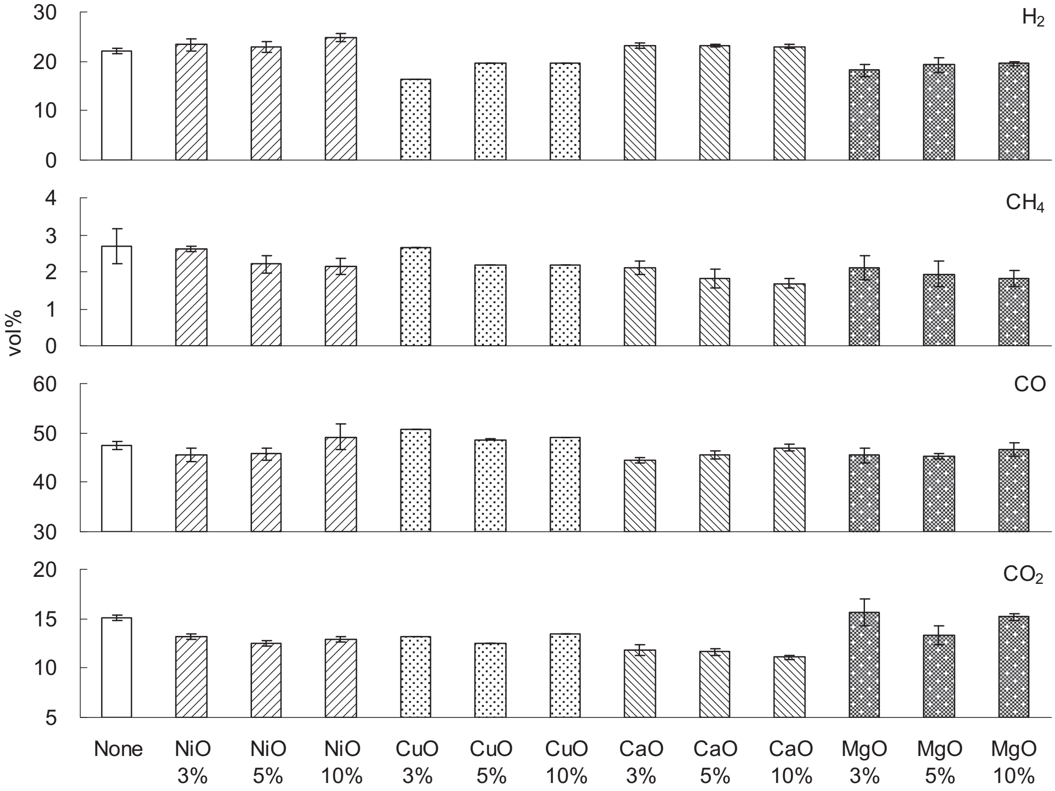

Fig. 3. The volumetric concentrations of H_2, CH_4, CO, and CO_2 in gaseous products.

Table 3
The molecules of H_2, CH_4, CO, and CO_2 produced with and without catalyst addition.

Catalyst	Molecules (mmole)			
	H_2	CH_4	CO	CO_2
None	21.63	2.63	46.51	14.74
NiO 10%	23.27	2.02	46.15	12.10
CuO 10%	17.51	1.96	43.88	12.07
CaO 10%	23.23	1.70	47.55	11.25
MgO 10%	18.36	1.70	43.97	14.27

addition, the relative weight percentages of gaseous, liquid, and solid products were about 58, 23, and 19 wt%, respectively. The average percentages of gaseous products under the addition of NiO, CuO, CaO, and MgO were about 52, 53, 62, and 65 wt%, respectively. Besides, the average percentages of liquid products under the addition of NiO, CuO, CaO, and MgO were about 32, 33, 24, and 18 wt%, respectively. The catalytic effect on the amount of solid residues was not as much as that on the productions of gas and liquid. Generally, the addition of either CaO or MgO increased the gaseous fraction, and the addition of either NiO or CuO increased the liquid fraction. The catalysts CaO and MgO can react with water to produce alkaline aqueous, which could be useful for H_2 production and thus to increase the gaseous yield. Besides, according to the work of Sui et al. (2012), the addition of sulfuric acid can promote the liquid yield of bagasse pyrolysis at low temperatures. The changes in gaseous and liquid fractions may imply that there could be two competitive reaction mechanisms which are the so-called Broido–Shafizadeh model (Bradbury et al., 1979; Varhegyi et al., 1994). Besides, the Fischer–Tropsch synthesis and the interactions among the product gases could influence the product fractions as well.

Although the maximum temperature for CaO addition was less than other metal oxides by about 10 °C and less than that without any addition by about 36 °C (Table 1), this difference was not in accordance with their product distributions. Generally, higher temperatures should be able to provide higher conversions (i.e., lower solid yield and higher gas and liquid yields).On the contrary, the conversion of CaO was higher than those of other metal oxides, and the conversion without any addition was the least. This may imply that nevertheless the temperature can affect the biomass pyrolysis a lot, there could be some other factors to change the product distribution, such as catalytic effects and the interactions among biomass components and evolved compounds.

The correlation coefficients of catalyst quantities and product yields were calculated to determine the correlation between them. The NiO quantity was medium positive correlated with liquid yield while was weak negative or strong negative correlated with gas or solid yield. The CuO quantity was medium positive correlated with liquid yield while was medium negative correlated with either gas or solid yield. The CaO quantity was strong negative correlated with solid yield while was medium positive or strong positive correlated with liquid or gas yield. The MgO quantity was medium positive or strong negative correlated with gas or liquid yield while its correlation with solid yield was negligible. Generally, the solid yield was slightly decreased with the increasing catalyst quantity. The liquid yield was significantly increased with the addition of either NiO or CuO. Adding either CaO or MgO increased the gas yield, while adding either NiO or CuO decreased it. However, it is still difficult to precisely define the catalytic effect on the product distribution. This could be owing to the complexity and sensitivity of microwave pyrolysis, especially when a single-mode microwave oven was used.

4.1. Kinetic analysis

The reaction kinetics of biomass pyrolysis by conventional methods has been researched in a great deal of literature, but kinetic analyses with respect to microwave pyrolysis of biomass are very few. Thus, this study tried to analyze the reaction kinetics of microwave pyrolysis of sugarcane bagasse with and without catalysts. By using the Arrhenius equation, the rate equation of biomass pyrolysis can be expressed as:

$$\frac{d\alpha}{dt} = k \cdot (1-\alpha)^n = A \cdot \exp\left(-\frac{E_a}{RT}\right) \cdot (1-\alpha)^n \tag{2}$$

where k is reaction rate, A is pre-exponential factor, E_a is activation energy, R is universal gas constant, n is the order of reaction, t is time, T is temperature, and α is the conversion ratio of biomass:

$$\alpha = \frac{m_0 - m_t}{m_0 - m_f} \tag{3}$$

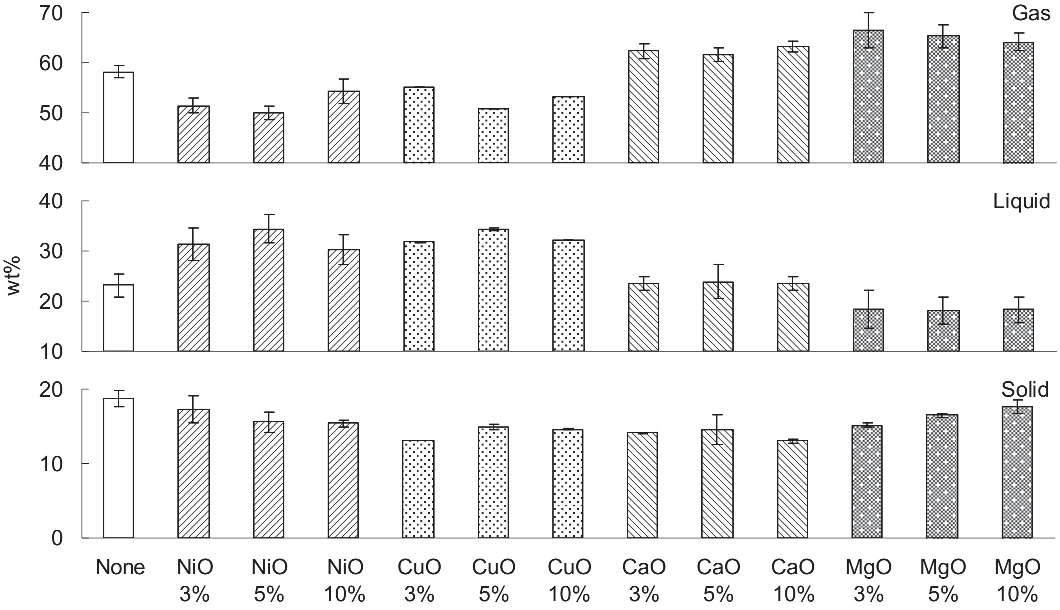

Fig. 4. Three-phase product distribution.

Table 4
The kinetic parameters of microwave pyrolysis of sugarcane bagasse with and without catalyst addition.

Catalyst	Activation energy, E_a (kJ/mol)	Pre-exponential factor, A (1/s)	Reaction rate, k (1/s)	Coefficient of determination, R^2
None	18.91	0.175	3.98×10^{-3}	0.99
NiO 10%	20.24	0.247	4.05×10^{-3}	0.99
CuO 10%	20.85	0.289	4.03×10^{-3}	0.99
CaO 10%	21.39	0.337	4.11×10^{-3}	0.99
MgO 10%	19.95	0.225	3.88×10^{-3}	0.99

where m_0 is the initial mass of biomass sample, m_t is the mass at any time, and m_f is the final mass at the end of reaction. By using a constant heating rate β (= dT/dt), Eq. (2) can be rearranged to:

$$\frac{d\alpha}{dT} = \frac{A}{\beta} \cdot \exp\left(-\frac{E_a}{RT}\right) \cdot (1-\alpha)^n \tag{4}$$

This study assumed that the biomass pyrolysis is a first-order reaction, so n is 1. According to the Coats–Redfern integral method (Coats and Redfern, 1964), Eq. (4) can be integrated and rearranged to:

$$\ln\left[\frac{-\ln(1-\alpha)}{T^2}\right] = \ln\left[\frac{AR}{\beta E_a}\left(1 - \frac{2RT}{E_a}\right)\right] - \frac{E_a}{RT} \tag{5}$$

From the linear regression of the left-hand-side term of Eq. (5) versus 1/T, the activation energy and pre-exponential factor can be determined. Table 4 lists the kinetic parameters of microwave pyrolysis of sugarcane bagasse at the catalyst addition ratio of 10 wt%. The coefficients of determination (R^2) of these linear regressions were all over 0.99. It can be seen that the catalyst addition slightly increased the activation energy (by 5–13%) but greatly increased the pre-exponential factor (by 28–92%). Higher pre-exponential factor can contribute to higher reaction rate, which can be seen in Table 4. Among these catalysts, CaO showed the biggest catalytic effect while MgO showed the smallest. Consequently, adding NiO, CuO, and CaO increased the reaction rate by 1.7%, 1.3%, and 3.3%, respectively, but adding MgO decreased it by 2.5%. In fact, this kinetic analysis is not as satisfactory as it should be due to the difficulty in acquiring precise relationship between biomass weight and temperature (i.e., continuous thermogravimetric data). To satisfy this demand, further adjustment of experimental apparatus should be necessary. Besides, traditional thermogravimetric analysis (TGA) could be used to simulate the microwave pyrolysis by controlling its temperature profiles to be similar to those of microwave heating.

5. Conclusions

With catalyst addition, many reaction results such as mass reduction ratio, calorific value of solid residues, and reaction rate can be increased using microwave heating. Either CaO or MgO addition can enhance the gaseous production, and either NiO or CuO addition can enhance the liquid production. Several secondary reactions including self-gasification and interactions among the gases originally produced during the pyrolysis stage could occur to change the composition of gaseous product and the final three-phase product distribution. Further researches about the effects of catalyst characteristics, biomass conditioning, and reaction atmosphere are still necessary to promote the maturity of the technology.

Acknowledgements

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